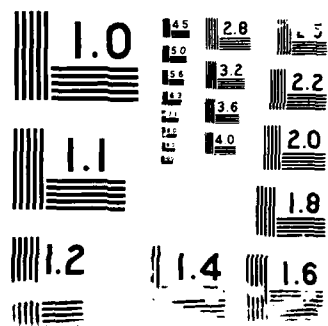


AD-A194 307 FEMTOGRAM LEVEL DETERMINATION OF COBALT AND CHROMIUM 1/1
BY LUMINOL CHEMILUM. (U) ARIZONA UNIV TUCSON DEPT OF
CHEMISTRY R D JALKIAN ET AL. 04 FEB 88 TR-63
UNCLASSIFIED N00014-86-K-0316 F/G 7/2 NL





SECUR

AD-A194 307

(4) 2

RT DOCUMENTATION PAGE

1a. REPORT SECURITY CLASSIFICATION UNCLASSIFIED			1b. RESTRICTIVE MARKINGS DTIC FILE 002	
2a. SECURITY CLASSIFICATION AUTHORITY			3. DISTRIBUTION / AVAILABILITY OF REPORT Approved for public release; distribution unlimited	
2b. DECLASSIFICATION / DOWNGRADING SCHEDULE				
4. PERFORMING ORGANIZATION REPORT NUMBER(S) 63			5. MONITORING ORGANIZATION REPORT NUMBER(S)	
6a. NAME OF PERFORMING ORGANIZATION University of Arizona		6b. OFFICE SYMBOL (If applicable)	7a. NAME OF MONITORING ORGANIZATION Office of Naval Research	
6c. ADDRESS (City, State, and ZIP Code) Department of Chemistry Tucson, Arizona 85721			7b. ADDRESS (City, State, and ZIP Code) Arlington, Virginia 22217	
8a. NAME OF FUNDING / SPONSORING ORGANIZATION Office of Naval Research		8b. OFFICE SYMBOL (If applicable)	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER N00014-86-K-0316	
8c. ADDRESS (City, State, and ZIP Code)			10. SOURCE OF FUNDING NUMBERS	
			PROGRAM ELEMENT NO.	PROJECT NO.
11. TITLE (Include Security Classification) "Femtoprogram Level Determination of Cobalt and Chromium by Luminol Chemiluminescence Detected by a Charge Coupled Device"				
12. PERSONAL AUTHOR(S) Rafi D. Jalkian and M. Bonner Denton				
13a. TYPE OF REPORT Technical		13b. TIME COVERED FROM 5/15/86 TO 4/30/89		14. DATE OF REPORT (Year, Month, Day) February 4, 1988
15. PAGE COUNT				
16. SUPPLEMENTARY NOTATION Prepared for presentation at the Pittsburgh Conference, New Orleans, Louisiana, February 22, 1988				
17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number) Chemiluminescence, luminol, 3-aminophthol hydrazide, cobalt (II), chromium (III), hydrogen peroxide, CCD, charge-coupled device, charge transfer device	
FIELD	GROUP	SUB-GROUP		
19. ABSTRACT (Continue on reverse if necessary and identify by block number) Recently, chemiluminescence analysis of low concentration cobalt and chromium in natural waters has enjoyed much success over the more time consuming method of flameless atomic absorption spectrometry (FAAS), which consists of sample preconcentration by solvent extraction followed by FAAS. New solid-state multichannel array detectors offer greater sensitivity and better performance in many respects over currently available photon counting photomultiplier tubes. These detectors have the potential of greatly improving low light level spectroscopic measurements. In this study, a solid-state, integrating, two-dimensional charge-coupled device (CCD) was used to measure the chemiluminescence spectra and to quantitatively determine chromium, cobalt, and hydrogen peroxide by luminol chemiluminescence. The RCA SID501EX charge-coupled device used in this study has the desirable characteristics of low readout noise, low dark current, and high quantum efficiency. This quantum efficiency is approximately 55% at the luminol chemiluminescence maximum of 425 nm. (over)				
20. DISTRIBUTION / AVAILABILITY OF ABSTRACT <input checked="" type="checkbox"/> UNCLASSIFIED/UNLIMITED <input type="checkbox"/> SAME AS RPT. <input type="checkbox"/> DTIC USERS			21. ABSTRACT SECURITY CLASSIFICATION UNCLASSIFIED	
22a. NAME OF RESPONSIBLE INDIVIDUAL M. Bonner Denton			22b. TELEPHONE (Include Area Code) (602) 621-6352	
			22c. OFFICE SYMBOL	

19. Abstract (continued)

The chemiluminescence was measured using two experimental configurations. One employed a flat field polychromator, and a sample cell to obtain the spectra of luminol-peroxide-metal chemiluminescence. The very low intensity background emission spectra were also measured. The second instrumental configuration consisting of the CCD and a sample cell was employed for quantitative determinations.

Utilizing the integrating capability of the detector to collect the chemiluminescence emission over a period of a few minutes, reproducible sample introduction and mixing is no longer a critical factor. Thus, by eliminating the variation introduced through non-uniform introduction of sample and mixing, excellent sensitivity and reproducibility were obtained.

Picogram level detection limits for both Co(II) and Cr(III) are the result of low readout noise, low dark current, and high quantum efficiency. The instrument's linear dynamic range is 5 orders of magnitude with 20 μL sample injected in 100 μL alkaline luminol solution.

OFFICE OF NAVAL RESEARCH

Contract N00014-86-K-0316

R&T Code 4131012---03

Technical Report No. 63

Femtoqram Level Determination of Cobalt and Chromium by
Luminol Chemiluminescence Detected by a Charge Coupled Device

by

Rafi D. Jalkian and M. Bonner Denton

Prepared for Presentation at the
Pittsburgh Conference
New Orleans, Louisiana
February 22, 1988

Department of Chemistry
University of Arizona
Tucson, Arizona 85721

February 4, 1988

Accession For	
NTIS GRA&I	<input checked="" type="checkbox"/>
DTIC TAB	<input type="checkbox"/>
Unannounced	<input type="checkbox"/>
Justification	
By	
Distribution/	
Availability Codes	
Dist	Avail and/or Special
A-1	

Reproduction in whole or in part is permitted for
any purpose of the United States Government.



This document has been approved for public release
and sale; its distribution is unlimited.

88 4 25 065

FEMPTOGRAM LEVEL DETERMINATION OF COBALT AND CHROMIUM
BY LUMINOL CHEMILUMINESCENCE DETECTED BY A
CHARGE-COUPLED DEVICE

Rafi D. Jalkian and M. Bonner Denton

Department of Chemistry
University of Arizona
Tucson, AZ 85721

Index Headings: Chemiluminescence, luminol, 3-aminophthol hydrazide, cobalt (II), chromium (III), hydrogen peroxide, CCD, charge-coupled device, charge transfer device

ABSTRACT

Recently, chemiluminescence analysis of low concentration cobalt and chromium in natural waters has enjoyed much success over the more time consuming method of flameless atomic absorption spectrometry (FAAS), which consists of sample preconcentration by solvent extraction followed by FAAS.

New solid-state multichannel array detectors offer greater sensitivity and better performance in many respects over currently available photon counting photomultiplier tubes. These detectors have the potential of greatly improving low light level spectroscopic measurements. In this study, a solid-state, integrating, two-dimensional charge-coupled device (CCD) was used to measure the chemiluminescence spectra and to quantitatively determine chromium, cobalt, and hydrogen peroxide by luminol chemiluminescence.

The RCA SID501EX charge-coupled device used in this study has the desirable characteristics of low readout noise, low dark current, and high quantum efficiency. This quantum efficiency is approximately 55% at the luminol chemiluminescence maximum of 425 nm.

The chemiluminescence was measured using two experimental configurations. One employed a flat field polychromator, and a sample cell to obtain the spectra of luminol-peroxide-metal chemiluminescence. The very low intensity background emission spectra were also measured. The second instrumental configuration consisting of the CCD and a sample cell was employed for quantitative determinations.

Utilizing the integrating capability of the detector to collect the chemiluminescence emission over a period of a few minutes, reproducible sample introduction and mixing is no longer a critical factor. Thus, by eliminating the variation introduced through non-uniform introduction of sample and mixing, excellent sensitivity and reproducibility were obtained.

Femtoogram level detection limits for both Co(II) and Cr(III) are the result of low readout noise, low dark current, and high quantum efficiency. The instrument's linear dynamic range is 5 orders of magnitude with 20 uL sample injected in 100 uL alkaline luminol solution.

DL/1113/87/2

TECHNICAL REPORT DISTRIBUTION LIST, GEN

	<u>No. Copies</u>		<u>No. Copies</u>
Office of Naval Research Attn: Code 1113 800 N. Quincy Street Arlington, Virginia 22217-5000	2	Dr. David Young Code 334 NORDA NSTL, Mississippi 39529	1
Dr. Bernard Douda Naval Weapons Support Center Code 50C Crane, Indiana 47522-5050	1	Naval Weapons Center Attn: Dr. Ron Atkins Chemistry Division China Lake, California 93555	1
Naval Civil Engineering Laboratory Attn: Dr. R. W. Drisko, Code L52 Port Hueneme, California 93401	1	Scientific Advisor Commandant of the Marine Corps Code RD-1 Washington, D.C. 20380	1
Defense Technical Information Center Building 5, Cameron Station Alexandria, Virginia 22314	12 high quality	U.S. Army Research Office Attn: CRD-AA-IP P.O. Box 12211 Research Triangle Park, NC 27709	1
DTNSRDC Attn: Dr. H. Singerman Applied Chemistry Division Annapolis, Maryland 21401	1	Mr. John Boyle Materials Branch Naval Ship Engineering Center Philadelphia, Pennsylvania 19112	1
Dr. William Tolles Superintendent Chemistry Division, Code 6100 Naval Research Laboratory Washington, D.C. 20375-5000	1	Naval Ocean Systems Center Attn: Dr. S. Yamamoto Marine Sciences Division San Diego, California 91232	1

END

DATE

FILMED

8-88

DTIC